

## WHAT IS CLAIMED IS:

1. A method of cleaving an O-linked oligosaccharide from a glycoprotein, said method comprising the steps of  
contacting a composition comprising a glycoprotein, wherein  
5 the glycoprotein comprises O-linked oligosaccharides, with a solution comprising a borane-ammonia complex to form a mixture comprising the glycoprotein and the borane-ammonia complex;  
incubating the mixture for a period of time sufficient to cleave  
the linked oligosaccharides from the glycoprotein; and  
10 forming a mixture comprising oligosaccharide alditol products and deglycosylated protein by-products.
2. The method of claim 1 further comprising the step of separating at least one cleaved oligosaccharide product from the other oligosaccharide products.
3. The method of claim 1 further comprising the step of separating  
15 at least one cleaved oligosaccharide product from the protein by-products.
4. The method of claim 2 or 3 further comprising the step of analyzing the structure of the oligosaccharide product.
5. The method of claim 4 wherein the structure of the cleaved oligosaccharide is analyzed by mass spectrometry.
- 20 6. The method of claim 5 wherein the mass spectrometry method is selected from the group consisting of matrix-assisted laser desorption ionization mass spectrometry and matrix-assisted laser desorption ionization/time-of-flight mass spectrometry.
7. The method of claim 2 or 3 wherein the separation is achieved  
25 using a cation exchange resin.
8. The method of claim 2 or 3 wherein the separation is achieved using a hydrophobic resin.
9. The method of claim 2 or 3 wherein the separation is achieved using a cation exchange resin and a hydrophobic resin.
- 30 10. The method of claim 1 wherein the incubation step is performed a temperature of about 40°C to about 50 °C.

11. The method of claim 1 wherein the incubation step is performed a temperature of about 35°C to about 55 °C.
12. The method of claim 1 wherein the incubation step is performed a temperature of about 30°C to about 60 °C.
- 5 13. The method of claim 1 wherein the incubation step is performed a temperature of about 20°C to about 60 °C.